

Republic of South Africa EDICT OF GOVERNMENT

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SANS 5210 (2010) (English): Water - Nitrate and nitrite content



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SANS 5210:2010

Edition 2.3

SOUTH AFRICAN NATIONAL STANDARD

Water — Nitrate and nitrite content



SANS 5210:2010

Edition 2.3

Table of changes

| Change No. | Date | Scope | |
|------------|------|--|--|
| Amdt 1 | 2004 | Amended to change the designation of SABS standards to SANS standards and to update referenced standards. | |
| Amdt 2 | 2006 | Amended to update a referenced standard. | |
| Amdt 3 | 2010 | Amended to update referenced standards, and to correct values for total oxidized nitrogen in the table in 6.2. | |

Foreword

This South African standard was approved by National Committee SABS SC 147A, *Water – Water sampling and analysis*, in accordance with procedures of the SABS Standards Division, in compliance with annex 3 of the WTO/TBT agreement.

This document was published in April 2010.

This document supersedes SANS 5210:2006 (edition 2.2).

A vertical line in the margin shows where the text has been technically modified by amendment No. 3.

Water — Nitrate and nitrite content

1 Scope and field of application

This standard specifies a method of measuring the concentration of nitrate and nitrite nitrogen in water and in wastewater.

The method is conveniently applied after the determination of free and saline ammonia, using SANS 5217, since the residue in the distillation flask after the performance of SANS 5217 can be used as the test portion to be reduced with Devarda's alloy.

Nitrite nitrogen should be determined separately by using SANS 6777 if the concentrations of both nitrate and nitrite nitrogen are required.

Amdt 2

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

Amdt 1

SANS 111/ISO 835, Laboratory glassware – Graduated pipettes.

Amdt 1; amdt 3

SANS 112/ISO 648, Laboratory glassware – Single volume pipettes.

Amdt 1

SANS 115/ISO 385, Laboratory glassware – Burettes.

Amdt 1; amdt 3

SANS 128/ISO 1042, Laboratory glassware - One-mark volumetric flasks.

Amdt 1

SANS 3696/ISO 3696, Water for analytical laboratory use – Specification and test methods.

SANS 5217, Water – Free and saline ammonia content.

SANS 5219, Nitrite content of water.

Amdt 2

SANS 6777/ISO 6777, Water quality – Determination of nitrite – Molecular absorption spectrometric method.

Amdt 2

Edition 2.3

3 Principle

After the removal of ammonical nitrogen from the test portion by distillation under alkaline conditions, the nitrate and nitrite are reduced with Devarda's alloy, resulting in a quantitative conversion to ammonia that is distilled over with the distillate into a boric acid solution where an equivalent amount of borate is produced. The borate is titrated with a standard acid solution to the equivalence point, which is detected with a mixed indicator.

4 Reagents

NOTE Unless otherwise specified, only use water that complies with the requirements of SANS 3696 and reagents of analytical reagent grade.

- 4.1 The reagents listed in SANS 5217.
- **4.2** Devarda's alloy (an alloy of 50 % copper (Cu), 45 % aluminium (AI), and 5 % zinc (Zn), and containing less than 0.005 % (m/m) nitrogen (N)).
- 4.3 Nessler's reagent.

5 Apparatus

- **5.1** The apparatus listed in SANS 5217.
- **5.2** Glassware. Where applicable, only use burettes, pipettes and volumetric flasks that comply with the requirements for class A items as specified in SANS 111, SANS 112, SANS 115 and SANS 128, as relevant.

 Amdt 1; amdt 3

6 Procedure

NOTE Unless otherwise specified, only use water that complies with the requirements of SANS 3696.

- **6.1** If free and saline ammonia has been determined using SANS 5217, proceed to 6.9.
- **6.2** Select a suitable volume of sample, using the following table as a guide:

| 1 | 2 |
|-------------------------|-------------|
| Total oxidized nitrogen | Sample size |
| as N in mg/L | mL |
| 0 – 1 | 500 |
| 2 – 10 | 250 |
| 11 – 20 | 100 |
| 21 – 50 | 50,0 |
| 51 – 100 | 25,0 |

Amdt 3

NOTE The sample size shown above is an estimate made on the basis of a titration using the standard sulfuric acid (H_2SO_4) solution $c(1/2 H_2SO_4) = 0.02 \text{ mol/L}$.

6.3 Transfer the aliquot to a distillation flask and dilute the sample to approximately 500 mL with water.

- **6.4** Add 5-6 drops of the bromothymol blue indicator solution and adjust the pH to a value in the range pH 6,0 (indicator yellow) to pH 7,4 (indicator blue), using either the sodium hydroxide (NaOH) solution or the sulfuric acid (H_2SO_4) solution, as appropriate.
- **6.5** Add 0,3 g of light magnesium oxide, mix and add some anti-bump beads.
- **6.6** Attach the distillation flask to the anti-splash head and connect to the condenser.
- **6.7** Distil vigorously until 275 mL ± 25 mL of distillate has been collected in a receiving flask.
- **6.8** Check the last few distillate volumes of 15 mL \pm 5 mL with Nessler's reagent (see 4.3) to ensure that all ammonia has been distilled over, and discard the distillate.
- **6.9** Allow the residue in the flask to cool.
- **6.10** Dilute to approximately 500 mL, add 1 g of Devarda's alloy (see 4.2) and mix.
- **6.11** Attach the distillation flask to the anti-splash head and connect to the condenser.
- **6.12** Immerse the condenser outlet in 50 mL of the boric acid/indicator solution in a receiving flask.
- **6.13** Boil the mixture gently for approximately 5 min to avoid excessive frothing, and then boil vigorously until 250 mL to 300 mL of distillate has been collected.
- **6.14** Titrate the distillate in the boric acid/indicator solution to the purple end-point, using the standard H_2SO_4 volumetric solution, $c(1/2 H_2SO_4) = 0.02 \text{ mol/L}$.

NOTE The standard H_2SO_4 volumetric solution, $c(1/2 \ H_2SO_4) = 0.10 \ mol/L$, may be used for titration of distillates from samples having high nitrate/nitrite contents.

6.15 Always carry a blank through the whole procedure, substituting water for the sample.

7 Expression of results

The nitrate and nitrite content, expressed as nitrogen (N) in mg/L, is given by the formula

$$\frac{(V_1 - V_2) \times c \times 14,01 \times 1000}{V_0}$$

where

 V_0 is the volume of the test portion, in millilitres;

- V_1 is the volume of sulfuric acid solution used in titration of the sample distillate, in millilitres;
- V_2 is the volume of sulfuric acid solution used in titration of the blank distillate, in millilitres;
- is the exact concentration of the sulfuric acid used in the titration, $c(1/2 \text{ H}_2\text{SO}_4)$, in moles per litre.

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SABS - Standards Division

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South African National Standards are updated by amendment or revision. Users of South African National Standards should ensure that they possess the latest amendments or editions.

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